

CLAIMS

1. A process for the production of metal fluorides comprising:
introduce a predetermined weight of anhydrous hydrofluoric acid into a reaction vessel and initiate a mixing action;
preheat a predetermined weight of anhydrous metal to a predetermined reaction temperature;
introduce aliquots of the anhydrous metal into the reaction vessel at intervals until the entire predetermined weight of the anhydrous metal has been added;
remove excess anhydrous hydrofluoric acid from the reaction vessel; and
remove a metal fluoride resultant product from the reaction vessel.
2. The process of claim 1 wherein the mixing action is selected from the group consisting of:
rotation,
stirring and
agitation.
3. The process of claim 1 wherein the anhydrous metal is a metal compound.
4. The process of claim 1 wherein the predetermined weight of anhydrous metal is introduced into the reaction vessel through a plunger device and port designed for such purpose.
5. The process of claim 1 further comprising:
exhausting an internally generated gaseous resultant product.
6. The process of claim 5 wherein the internally generated gaseous resultant product is exhausted through an automatic regulating gas back-pressure valve.
7. The process of claim 1 wherein, after the reactants have been fully introduced into the reaction vessel, the reaction vessel is maintained at a predetermined reaction pressure and temperature for a minimum period of time.
8. The process of claim 7 wherein the minimum time is four hours.

9. The process of claim 1 wherein remove the excess anhydrous hydrofluoric acid is removed from the reaction vessel by evaporating the excess acid through a gas backpressure valve.

10. The process of claim 1 further comprising:
place the resultant metal fluoride product in an appropriately designed and constructed open container; and
place the container and its contents in an oven capable of maintaining an inert environment while heating the metal fluoride.

11. The process of claim 10 further comprising:
heating the resultant metal fluoride at $95^{\circ}\text{C} \pm 4^{\circ}\text{C}$ for a period of approximately two hours.

12. The process of claim 11 further comprising:
after heating the metal fluoride at $95^{\circ}\text{C} \pm 4^{\circ}\text{C}$, bring the temperature of the metal fluoride to within 10°C of the metal fluoride's decomposition or melting point, whichever temperature is lower.

13. The process of claim 12 further comprising:
cooling the metal fluoride to ambient temperature in a sealed desiccator that is free of moisture and stray gases.

14. The process of claim 1 wherein the reaction vessel capable of withstanding exposure to anhydrous hydrofluoric acid and capable of operating under internal system working pressures in the range of zero to 400 psia and temperatures in the range of -200°F to 300°F .

15. The process of claim 14 wherein the reaction vessel is equipped with an automatic regulating gas back pressure valve, settable at back pressures ranging from zero psia to 400 psia.

16. The process of claim 14 wherein the reaction vessel is equipped with a plunger-type device that allows solid, granular reactant materials to be introduced to the reaction vessel, while the reaction vessel is under vacuum or pressure, without allowing fluids to escape from or enter into the reaction vessel.

17. The process of claim 1 further comprising:
purging the reaction vessel a minimum of three successive times with pure nitrogen gas;
and
filling the reaction vessel with pure nitrogen gas to the pressure at which it is intended to conduct the reaction.

18. The process of claim 1 wherein the aliquots are 10% of the entire predetermined weight of the anhydrous metal.

19. The process of claim 1 wherein the aliquots are added using a plunger-type device that allows solid, granular reactant materials to be introduced to the reaction vessel, while the reaction vessel is under vacuum or pressure, without allowing fluids to escape from or enter into the reaction vessel.

20. The process of claim 1 wherein the weight ratio of the anhydrous hydrofluoric acid to anhydrous metal is a multiple of the stoichiometric combining weight of the metal reactant.

21. The process of claim 20 wherein the weight ration is not less than 2 and not greater than 60.

22. The process of claim 20 further comprising:
determining an optimum weight ratio comprising:
producing batches of the metal fluoride at various ratios; and
rating the resultant metal fluoride product by its suitability for an intended application of such product.

23. The process of claim 1 wherein the process of removing excess anhydrous hydrofluoric acid from the reaction vessel comprises:
progressively reducing a set pressure on a gas backpressure valve, while maintaining a temperature above 19.8°C on the reaction vessel, until all of the anhydrous hydrofluoric acid has volatilized.

24. The process of claim 23 further comprising:
passing the volatilized vapor phase anhydrous hydrofluoric acid through a heat exchanger to reduce the temperature below the condensation temperature at standard atmospheric pressure;
and
recovering and condensing the anhydrous hydrofluoric acid for use in the process again.
25. The process of claim 1 wherein the anhydrous metal is less than anhydrous or not anhydrous.
26. The process of claim 1 wherein the anhydrous metal is less than essentially chemically pure or not chemically pure.
27. The process of claim 1 wherein the anhydrous hydrofluoric acid is less than completely anhydrous or is not anhydrous.
28. The process of claim 1 wherein the reaction vessel is set at a temperature other than the predetermined reaction temperature.
29. The process of claim 1 wherein the mixing action is not engaged.
30. The process of claim 1 wherein the anhydrous metal is preheated to a temperature other than the reaction temperature.

31. A process for the production of metal fluorides comprising:
providing hydrofluoric acid in a reaction vessel;
introducing aliquots of a metal reactant into the reaction vessel at intervals until a predetermined weight of the metal has been added, wherein the weight ratio of the hydrofluoric acid to metal is a multiple of a stoichiometric combining weight of the metal;
agitating the hydrofluoric acid and metal reactants in the reaction vessel;
venting excess hydrogen chloride gas generated during a reaction between the hydrofluoric acid and metal reactants; and
maintaining the hydrofluoric acid and metal reactants at a predetermined pressure and predetermined temperature for a minimum period following the introduction of the metal reactants.

32. The process of claim 31 further comprising:
removing a resultant metal fluoride product from the reaction vessel;
heating the metal fluoride product; and
placing the metal fluoride product in a desicator.

33. The process of claim 31 wherein the hydrofluoric acid and metal reactant are anhydrous.

34. A process for producing ferric trifluoride comprising:
providing hydrofluoric acid in a reaction vessel;
introducing ferric trichloride into the reaction vessel at intervals until a weight ratio of the anhydrous hydrofluoric acid to the ferric trifluoride is between 2 and 60;
agitating the hydrofluoric acid and ferric trichloride in the reaction vessel;
venting excess hydrogen chloride gas generated during a reaction between the hydrofluoric acid and ferric trichloride;
removing excess anhydrous hydrofluoric acid from the reaction vessel; and
removing a ferric trifluoride resultant product from the reaction vessel.